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PERMEABILITY OF FLEXIBLE MATERIALS USED IN FUEL STORAGE
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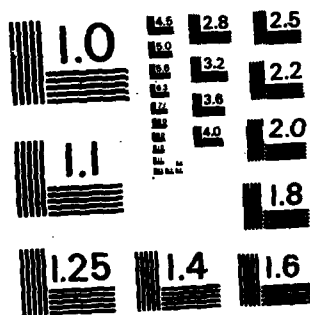
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TECHNICAL NOTE
MRL-TN-460

PERMEABILITY OF FLEXIBLE MATERIALS USED IN FUEL STORAGE TANKS

PART II - A METHOD OF MEASUREMENT AND PRELIMINARY RESULTS

Robert J. Mathers

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**DEPARTMENT OF DEFENCE
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TECHNICAL NOTE

MRL-TN-460

PERMEABILITY OF FLEXIBLE MATERIALS USED IN FUEL STORAGE TANKS

PART II - A METHOD OF MEASUREMENT AND PRELIMINARY RESULTS*

Robert J. Mathews

ABSTRACT

A method has been developed for the determination of the permeability of flexible tank fabrics to liquid fuels. The method, which employs a mass spectrometer, enables the permeabilities of fabrics at 20°C and at elevated temperatures to be assessed using a test liquid of 70% iso-octane and 30% toluene. Test results have been compared with results obtained using the standard gravimetric method and recommendations have been made for future work.

- * Permeability of Flexible Materials Used in Fuel Storage Tanks
Part I - General Review
MRL-TN-459 by B.C. Ennis

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ABSTRACT

A method has been developed for the determination of the permeability of flexible tank fabrics to liquid fuels. The method, which employs a mass spectrometer, enables the permeabilities of fabrics at 20°C and at elevated temperatures to be assessed using a test liquid of 70% iso-octane and 30% toluene. Test results have been compared with results obtained using the standard gravimetric method and recommendations have been made for future work.

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PERMEABILITY OF FLEXIBLE MATERIALS

USED IN FUEL STORAGE TANKS

PART II. A METHOD OF MEASUREMENT AND PRELIMINARY RESULTS

1. INTRODUCTION

A suitable method for monitoring the permeability of flexible POL tank fabric to hydrocarbon fuels over a range of temperatures was required [1] because of the occurrence of serious leakage problems with POL tanks, especially when the tanks were required to store MT gasoline in Australian tropical areas.

The existing (gravimetric) test method (MIL-T-6396C) for the permeability of POL tank fabric measures the loss of weight of permeant in a test rig during an eight day period [2]. This method is unsatisfactory because of the difficulty frequently experienced in obtaining adequate sealing between the fabric and the test rig; any leakage gives an inflated value of permeability [3].

This report contains details of a new method developed at MRL for the measurement of the permeability of POL fabrics, together with some preliminary results.

2. REQUIREMENTS OF THE METHOD

The requirement was to develop a sensitive instrumental method to monitor the permeability of POL tank fabrics to test liquids. It was necessary that the test specimen be actual POL tank fabric, preferably of similar size to that required by MIL-T-6396C, that is a disc of 50.8 mm diameter, and that the method should be capable of being used over the temperature range that tanks in service may experience in the tropical areas of Australia. During the trial of a 30,000 gallon (135,000 L) POL tank at JTTRE, Innisfail, North Queensland, skin temperatures greater than 60°C were recorded [4].

The specification required that the POL tank fabric have a maximum permeability (flux) of $8 \text{ mL/m}^2/\text{day}$ at 25°C [3]. This corresponds to a flow of approximately $1.6 \times 10^{-2} \text{ mg/min}$ through a sample of fabric having a diameter of 65 mm, assuming the bulk density of the permeant is equal to that of toluene. If the air-flow through the cell was 1 L/min , the concentration of permeant would be 16 mg/m^3 .

It would be possible to analyse this concentration of permeant directly by coupling the air flow from the cell to either a mass spectrometer fitted with a membrane inlet system, or a plasma chromatograph [5]. MRL does not have access to either of these systems.

Alternatively, the permeant may be trapped in a suitable receiver downstream from the cell and subsequently analysed by gas-liquid-chromatography (GLC) or mass spectrometry (MS). The merits of GLC and MS methods of analysis are discussed in Appendix 1.

The initial requirement was to analyse the test liquid containing 70% iso-octane and 30% toluene. However, there was a possibility that a later requirement for testing the permeability of POL tanks in the field would arise. The liquid in this situation would be MT gasoline, which contains many components and would have been difficult to analyse by conventional GLC (Section 4.7). However, the analysis of the complex MT gasoline in terms of hydrocarbon types (for example, alkanes, cycloalkanes, alkylbenzenes) would have been relatively simple by MS.

Therefore, mass spectrometry was chosen as the analysis technique for the preliminary measurements because of the higher sensitivity of the MS and the less complex analytical data for MT gasoline.

3. EXPERIMENTAL PROCEDURES

3.1 Apparatus

A flow-through cell was designed and constructed by making simple modifications to used spare parts from the vacuum system of the Atlas CH4 mass spectrometer (Fig. 1). The diameter of the fabric exposed in this cell was 65 mm. The cell and collection system were assembled as shown in Fig. 2, in an air-conditioned room ($20 \pm 2^\circ\text{C}$).

The initial procedure was to flush the cell with dry nitrogen (CIG Industrial Grade) at 1 L/min flow-rate for one hour, and then connect the cell to the cold trap and collect the permeant for 30 minutes. Liquid nitrogen

was used to cool the cold trap, which was packed with silanised glass wool (100 mm deep, approximately 10.5 g) (Note 1).

3.2 Transfer of Permeant from Cold Trap to MS Tube

After the permeant had been collected, the cold trap was transferred to the system shown in Fig. 3. PTFE (Teflon) taps were used in this system to avoid the loss of permeant that would have occurred if greased taps had been used. After the system had been evacuated to a pressure of 1.3×10^{-5} Pa (1 mm Hg), the flask of liquid nitrogen was removed from the trap and used to cool the MS tube. The cold trap was then heated. A transfer time of one hour was found to be necessary to ensure that more than 99% of the permeant had been transferred from the cold trap tube to the MS tube. The MS tube was then connected to the MS and the permeant analysed as outlined in Appendix 2.

3.3 Experiments at Elevated Temperatures

The glass connection tube between the cell and cold trap (Fig. 2) was replaced by a glass U-tube which allowed the complete cell to be immersed in a thermostatted water bath. The dry nitrogen flowing through the cell was preheated by passing it through a 1 metre length of coiled copper tubing (o.d. = 6.4 mm, i.d. = 3.2 mm) immersed in the water bath. The permeability was then determined as outlined in Sections 3.1 and 3.2.

4. RESULTS AND DISCUSSION

4.1 POL Fabric A

A piece of POL tank fabric (Fabric A) which had already been shown (by the MIL-T-6396C method) to have a permeability above the specification of 8 mL/m²/day at 25°C [6], was investigated. After 8 and 11 days of soaking in the test liquid (Table 1), the total permeability was 11.3 mL/m²/day and 11.5 mL/m²/day. This is consistent with the result obtained with the MIL-T-6396C method.

Using the newly developed analytical technique, it was found that the permeation ratio of toluene to iso-octane was approximately 9 to 2.

NOTE 1 The trapping efficiencies of silanised glass wool, ceramic insulator beads, glass helices and 3 mm diameter glass balls were checked by using a second cold trap in series with the first. A silanised glass wool trap resulted in 99% efficiency. The other materials all had trapping efficiencies less than 95%. The amount of permeant collected was found to be independent of flow rates between 0.5 and 10 L/min.

Taking into account the ratio of the components of the test liquid (70% iso-octane, 30% toluene), the permeation of toluene was approximately 10 times greater than that of iso-octane through Fabric A. Similar behaviour was observed with Fabric B, particularly at elevated temperatures (Table 6).

4.2 POL Fabric B

Pieces of fabric (Fabric B) were obtained from a large, flexible, POL tank that had been in service with the Australian Army in North Queensland. One piece was taken from the top section of the tank (Sample B1), and two pieces were taken from different side sections of the same tank (Samples B2 and B3). Permeability data for the fabrics are given in Tables 2-6.

From the plot of log permeability against $1000/T$ ($^{\circ}K$) (Section 4.4 and Fig. 5) the permeability of this fabric to both iso-octane and toluene is approximately 1.4 times greater at $25^{\circ}C$ than $20^{\circ}C$. Therefore, based on the permeability results obtained at $20^{\circ}C$ for the pieces of fabric within 9 days of the start of soaking (Tables 2, 3 and 4), all of the test pieces had permeabilities under the specified limit of $8 \text{ mL/m}^2/\text{day}$ at $25^{\circ}C$. This agrees with the results obtained with the MIL-T-6396C method for fabric from the same tank [6].

However, the results obtained for Fabric B for periods longer than 9 days indicate that the permeability of the fabric increased with the length of time that the fabric was left soaking in the cell (Tables 2 and 4). As the period of service of a POL tank would normally be much longer than 9 days, these results raise serious questions concerning the relevance of the MIL-T-6396C method.

4.3 Seam from Tank (Fabric B)

Because of problems associated with the failure of seams in flexible tanks, permeability studies were undertaken with samples taken from these specific areas. The seam tested, which contained three layers of fabric, had a permeability of $0.5 \text{ mL/m}^2/\text{day}$ after four days of soaking (Table 5). This result was well within the specification limit.

However, when tested after 20 days of soaking, the permeability had increased to greater than $8 \text{ mL/m}^2/\text{day}$, and was still increasing.

4.4 POL Fabric B at Elevated Temperatures

After 15 days at $20^{\circ}C$ in a permeability cell, a fabric sample had a measured permeability of $0.2 \text{ mL/m}^2/\text{day}$. The complete cell was then immersed in a waterbath (Section 3.3) in order that the effect of temperature changes on the rate of permeability could be investigated.

On each working day, the waterbath containing the cell was cycled from 20°C to 60°C (over a period of 4 hours) and then allowed to cool. On a number of days the permeability of the fabric was determined at 20°C and/or 60°C, and on two days the permeability was determined at 20, 30, 40, 50 and 60°C (Table 6). After 28 days the experiment had to be terminated because the adhesive holding the filling funnel to the cell began to fail, allowing water to enter the test cell.

Plots of the amounts of toluene and iso-octane permeating at 60°C against time (Fig. 4) indicated that after approximately 12 days, the permeability of iso-octane reached a constant value, whereas the permeability of toluene increased throughout the experiment. This is also illustrated by the graphs of $\log(\text{Permeability})$ against $1000/T$ (°K) (Fig. 5), using data from days 12 and 28 of Table 6.

Figure 5 also indicates that the permeability of the fabric to both toluene and iso-octane follows the Arrhenius equation. Therefore, if the permeability of a particular liquid through a piece of this fabric is known at a certain temperature, it should be possible to predict the permeability at other temperatures provided other conditions (for example, pressure on the tank walls) are independent of temperature.

For the fabric tested, the permeability at 60°C is more than ten times greater than the permeability at 20°C.

It should be noted that the conditions in this elevated temperature experiment, that is the temperature of the test liquid and air flowing over the fabric were approximately equal to the temperature of the fabric, may differ from the conditions experienced by a tank in service.

4.5 Discussion of NS Test Method

The major apparent drawback with the described method was the poor reproducibility of some permeability values, even when the values were determined either twice on the same day, or on successive days. In one case these results varied by as much as $\pm 15\%$ (Days 6 and 7 in Table 2). However, as stated in Section 3.1, the temperature in the laboratory varied by $\pm 2^\circ\text{C}$. An increase in temperature from 18°C to 22°C should result in an increase in permeability of between 20% and 30% (Fig. 5). This variation in temperature is probably the major cause of the poor reproducibility of some of the results, and illustrates the requirement for a constant temperature laboratory (Note 2).

NOTE 2 At the time that these results were obtained, they were regarded as preliminary and it was thought that further experiments could be performed using selected fabrics under more suitable conditions. However, this project was terminated in favour of higher priority work before this could be arranged.

Some of the results obtained at elevated temperatures also showed variations of approximately 15% from the Arrhenius equation (Fig. 5). This was also probably a result of slight fluctuations ($\pm 2^\circ\text{C}$) in the temperature of the waterbath used in the particular experiments.

Other disadvantages with the MS method were:

- (i) The time required to transfer the trapped permeant from the cold trap to the MS tube (1 hour);
- (ii) The requirement for cryogenic liquid for the cold trap; and
- (iii) The requirement for a MS with a quantitative gas-liquid inlet system.

4.6 Comparison of Gravimetric and MS Test Methods

The specified gravimetric method [2] is of limited use for fabrics of low permeability, since small changes (10 mg or less) must be measured in a relatively large mass (about 400 g). In the present case, samples of Fabric B gave permeabilities between 0 and 4 mL/m²/day by the gravimetric method, and for two samples an apparent weight gain was recorded [6]. Further, the recorded weight changes frequently indicated a decreasing rate of weight loss as the test proceeded, suggesting that at least part of the loss was not by permeation through the fabric. At the level of permeability required of POL tank fabrics, the specified gravimetric method cannot readily distinguish between materials that may just pass or just fail.

In contrast, the present method will only measure the fluid that has permeated through the fabric (losses around the edges of the cell are not detected), and can be directly calibrated by injecting comparable amounts of permeant into the system. Both composition and quantity are measured, and the required equipment, particularly if GLC measurement is used, is more likely to be available in a quality assurance laboratory than a suitable balance.

4.7 Recommendations for Future Investigations

Since the completion of this investigation, a new type of GLC column which can separate and elute all of the alkanes in MT gasoline before the elution of aromatics [7] has become available at NRL. The use of a GLC with an automatic integration system and a column of this type would allow simple and rapid determination of total permeant and the ratio of alkane/aromatic content of the permeant, even for MT gasoline.

GLC Concentrator/Headspace injection systems, for example the SGE Unijector (7), have also become available recently. A Concentrator/Headspace injection system allows more convenient collection of the hydrocarbon sample than the bubbler technique.

4.6 General Discussion

POL tanks consist of composite layers of different polymeric materials that have been bonded together. The fabrics which were used in this investigation were obtained from tanks that had been in service in different parts of Australia. As well as variations between different batches of nominally the same fabric, there could be differences in the stress under storage and the amount of solar radiation and hence deterioration that different parts of the same tank may have experienced. Any of the above factors could account for the variations found in the permeability of the pieces of fabric (Samples B1, B2 and B3) after they had been soaking for similar periods in the cell (Tables 2, 3 and 4). A thorough investigation of the permeability of unused POL fabrics, their individual composite layers, and the adhesives used on the seams, would be a minimum requirement for the elucidation of mechanisms of permeation.

5. CONCLUSIONS

1. The test cell described in conjunction with MS analysis, gave results in general agreement with permeability determined by the MIL-T-6396C gravimetric method. The new method has several advantages in sensitivity and analytical capability which make it suitable for investigational work not possible with the existing specified method.
2. The results obtained indicate that with fabrics of very low permeability the standard methods may not allow sufficient time for the true permeability of the fabric to be determined.
3. The described test cell conveniently determines permeability at temperatures above ambient. Permeability follows the Arrhenius equation in the temperature range 20-60°C, for some fabrics, and the permeability can increase tenfold within this range. This is relevant to the use of flexible POL tanks in Australian tropical conditions.
4. The new method permits separate study of the aromatic and aliphatic components of the permeating test fluid.

6. RECOMMENDATIONS FOR FUTURE WORK

The results of the present study indicate that the following topics warrant further investigation:

1. The GLC-FID method should be investigated for use as the analytical technique.
2. Permeability data should be obtained for new (unused) fabrics and the individual components of the fabrics.

3. A thermostatted box rather than a waterbath should be used for future work.

7. ACKNOWLEDGEMENTS

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APPENDIX 1

GLC AND MS ANALYSIS

1. GLC Analysis

Conventional GLC analysis would require that the permeant vapour flowing through the cell be trapped in a bubbler tube containing a suitable nonvolatile liquid for example, diethylphthalate. The minimum volume of nonvolatile liquid required in a bubbler is 2 mL, and the maximum volume of the sample that can be injected into the GLC is 5 μ L. Using the GLC equipped with a flame ionisation detector (GLC-FID), the limit of detection of hydrocarbons is approximately 10 μ g/mL [8].

Assuming a collection time of 30 minutes, an area of permeation of 3318 mm² (for a cell with diameter of 65 mm), and the maximum permissible flux of 8 mL/m²/day at 25°C, the amount of permeant trapped is 0.48 mg. This corresponds to a concentration of 240 μ g/mL of permeant in nonvolatile liquid.

Therefore components of a fuel mixture at the level of 4% would just be detectable by GLC-FID. The sensitivity could be effectively increased by increasing the collection time. For example, subject to the separation characteristics of the GLC column, a 2 hour collection would allow a minor component at the level of 1% to be detectable by GLC-FID.

2. Mass Spectrometric Analysis

The Atlas CH4 mass spectrometer (MS) has a gas inlet system that enables the total amount of a volatile sample to be analysed. Under normal operating conditions (electron energy 70 eV, 3.0 kV applied to the secondary electron multiplier), a sample of 0.5 μ L of toluene gives a mass spectrum with the two major peaks (m/e 91 and 92) having intensities of 1.65 and 1.26 volts respectively (Appendix 2). With a mixture of two chemically dissimilar components, for example iso-octane and toluene (see Appendix 2) there is no overlap between major peaks. A peak of 1 mV may be measured with the Atlas CH4 MS (that is, noise level is approximately 0.5 mV). Therefore, the approximate detection limit for toluene and other hydrocarbons having similar molecular weights is approximately 3×10^{-4} μ L.

With an area of permeation of 3318 mm², a permeability of 8 mL/m²/day corresponds to 0.55 μ L of permeant collected in 30 minutes. Therefore, a component at the level of 0.5% would just be detectable by MS.

APPENDIX 2

CALCULATION OF PERMEABILITY FROM MS DATA

The two most intense peaks in the mass spectrum of toluene are at m/e 91 and 92. The two most intense peaks in the mass spectrum of *iso*-octane are at m/e 56 and 57. The peaks at m/e 91 and 92 in *iso*-octane are less than 0.01% of the base peak, and the peaks at m/e 56 and 57 in toluene are approximately 0.02% of the base peak. Therefore, the amounts of toluene and *iso*-octane in a sample containing only these two compounds can be determined by a simple calculation using the intensities of the peaks at m/e 56, 57, 91 and 92.

To calibrate the MS, the permeation cell was replaced with a syringe inlet, and a 0.5 μ L sample of either toluene or *iso*-octane was injected into the flow system. With a nitrogen flow of 1 L/min, all of the sample was transferred to the cold trap within 20 minutes. The sample was then transferred to the MS inlet tube and analysed.

Seven separate calibrations of both *iso*-octane and toluene were performed over a seven week period. For toluene, the mean value of the sum of peak heights at m/e 91 and 92 (ΣT) was 2.91 volts with extreme values of 2.74 and 3.03. For *iso*-octane, the mean value of the sum of peak heights at m/e 56 and 57 (ΣO) was 2.46 volts with extreme values of 2.35 and 2.55.

The amount of toluene that permeates a fabric ($\text{mL/m}^2/\text{day}$) can therefore be expressed by

$$\left(\frac{\Sigma T}{2.91}\right) \left(\frac{0.5}{10^3}\right) \left(\frac{10^6}{3318}\right) \left(\frac{48}{1}\right) = 2.49 \times \Sigma T$$

where ΣT is the sum of peak heights (in volts) of m/e 91 and 92 obtained for a 30 minute experiment. Likewise the amount of *iso*-octane that permeates ($\text{mL/m}^2/\text{day}$) is $2.94 \times \Sigma O$, where ΣO is the sum of peak heights (in volts) of m/e 56 and 57 obtained for a 30 minute experiment.

TABLE 1

PERMEABILITY OF FABRIC A AT 20°C

DAY	PERMEABILITY (mL/m ² /day)		
	Toluene	Iso-octane	Total
8	9.0	2.3	11.3
11	9.1	2.4	11.5

TABLE 2

PERMEABILITY OF FABRIC SAMPLE B1 AT 20°C

DAY	PERMEABILITY (mL/m ² /day)		
	Toluene	Iso-octane	Total
4	0.10	0.04	0.14
6	0.09	0.03	0.12
7	0.10	0.06	0.16
13	0.08	0.06	0.14
21	0.24	0.12	0.36
25	0.47	0.23	0.70

TABLE 3

PERMEABILITY OF FABRIC SAMPLE B2 AT 20°C

DAY	PERMEABILITY (mL/m ² /day)		
	Toluene	Iso-octane	Total
5	0.13	0.10	0.23
5	0.09	0.12	0.21
6	0.13	0.13	0.26

TABLE 4

PERMEABILITY OF FABRIC SAMPLE B3 AT 20°C

DAY	PERMEABILITY (mL/m ² /day)		
	Toluene	Iso-octane	Total
1	0.02	0.02	0.04
9	0.03	0.02	0.05
13	0.15	0.07	0.22
15	0.14	0.06	0.20
17*	0.24	0.08	0.32
25*	0.25	0.07	0.32
27*	0.52	0.09	0.61
33*	0.50	0.06	0.56
43*	1.49	0.09	1.58

* These values obtained at 20°C during the elevated temperature experiment (see Table 6).

TABLE 5

PERMEABILITY OF SEAM OF TANK (FABRIC B) AT 20°C

DAY	PERMEABILITY (mL/m ² /day)		
	Toluene	Iso-octane	Total
3	0.27	0.18	0.45
4	0.25	0.20	0.45
20	7.25	0.81	8.06
24	7.84	0.79	8.63

TABLE 6

PERMEABILITY OF FABRIC SAMPLE B3 AT ELEVATED TEMPERATURES

DAY	TEMP (°C)	PERMEABILITY (mL/m ² /day)		
		Toluene	Iso-octane	Total
0	20	0.14	0.06	0.20
2	20	0.24	0.08	0.32
9	60	1.92	0.41	2.33
10	20	0.25	0.07	0.32
(22	0.52	0.09	0.61
(31	0.97	0.16	1.13
12(41.5	1.67	0.32	1.99
(50	2.76	0.56	3.32
(60	5.48	1.32	6.80
18	20	0.50	0.06	0.56
	60	12.95	1.56	14.51
20	60	15.06	1.35	16.41
24	60	17.93	1.12	19.05
(20	1.49	0.09	1.58
(30	3.06	0.19	3.25
28(42	6.97	0.44	7.41
(51	13.20	0.74	13.94
(60	20.37	1.23	21.60

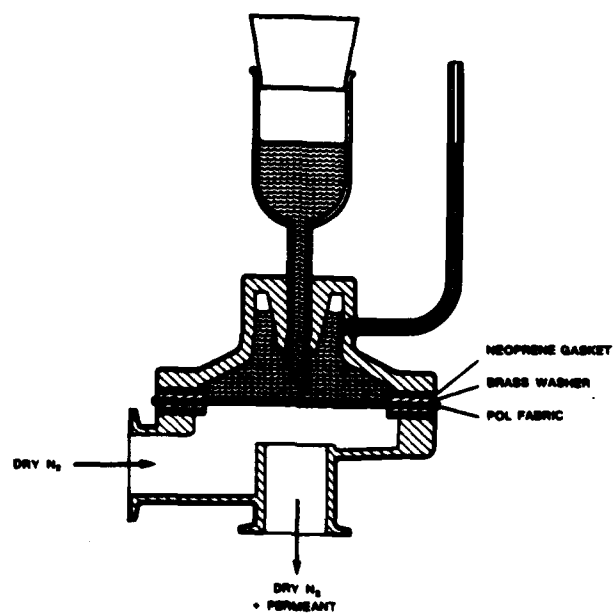


FIG. 1 Cross-section of Permeability Cell (Half Scale)

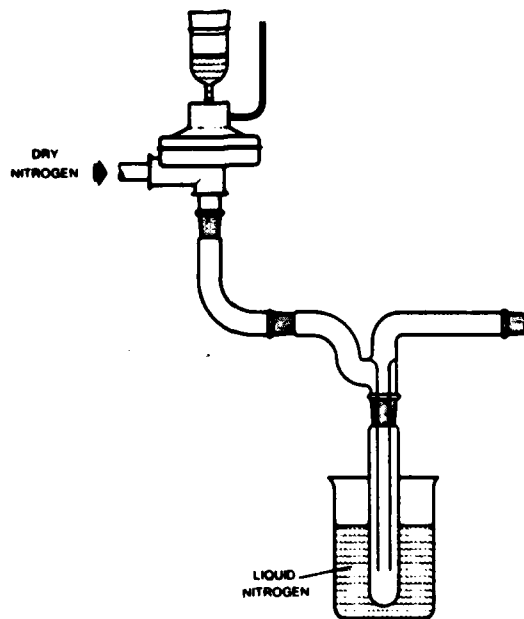


FIG. 2 Collection of Permeant in Cold Trap

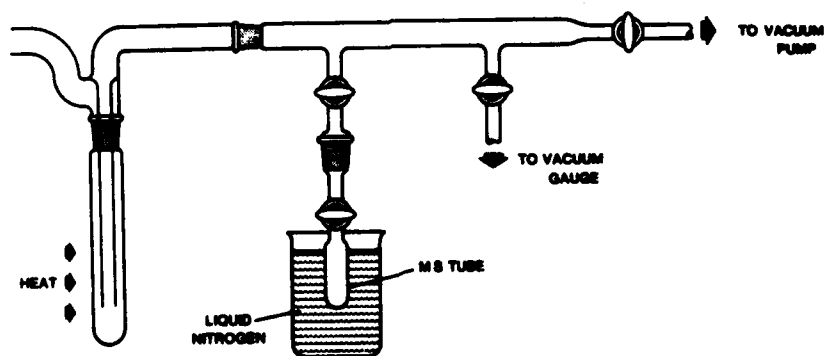


FIG. 3 Transfer of Permeant to MS Tube

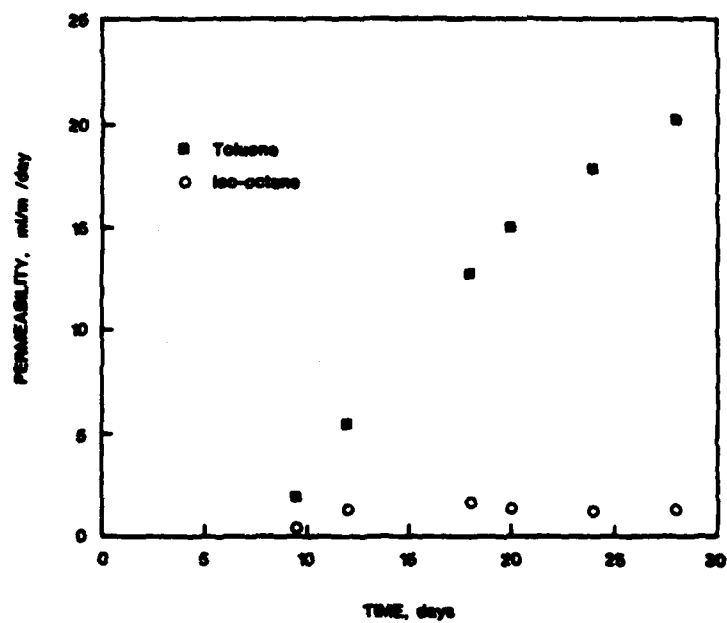


FIG. 4 Permeability of Toluene and Iso-octane at 60°C vs. Time

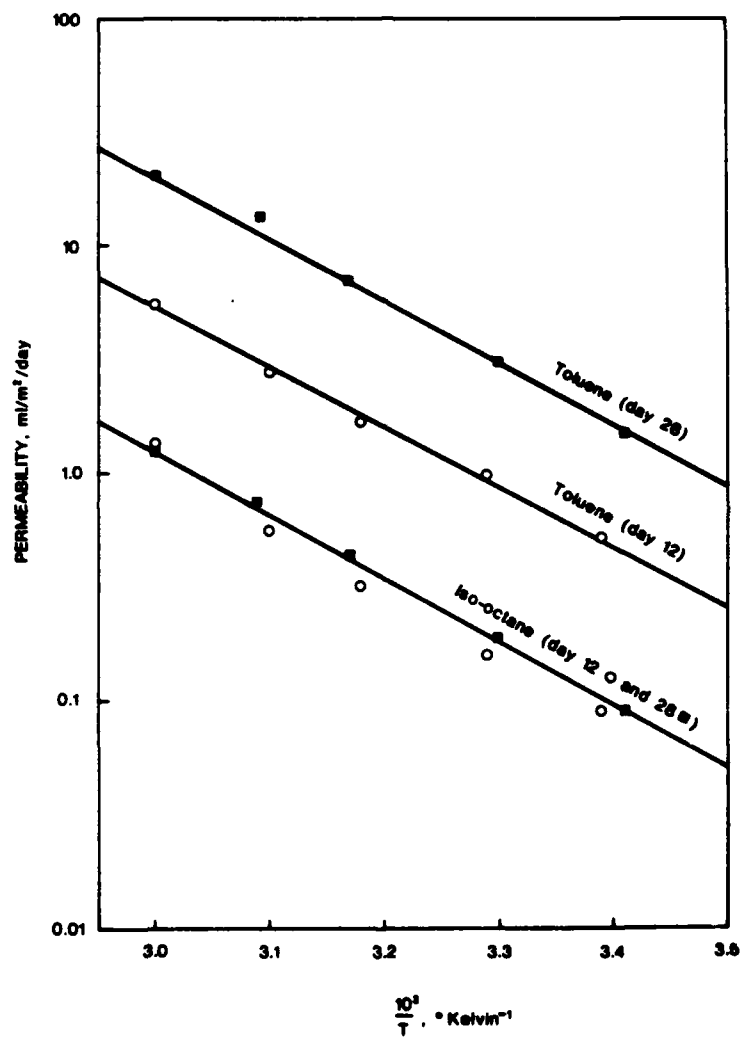


FIG. 5 Log (Permeability) vs. 1000/Temperature ($^\circ\text{K}$)

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